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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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(21) International Application Number: PCT/GB92/01527 (22) International Filing Date: 19 August 1992 (19.08.92) (30) Priority data: 9117913.5 20 August 1991 (20.08.91) GB (71) Applicant (for all designated States except US): SMITH & NEPHEW PLC [GB/GB]; 2 Temple Place, Victoria Embankment, London WC2R 3BP (GB). (72) Inventors; and (75) Inventors/Applicants (for US only) : O'REILLY, Angela, Noeleen [IE/GB]; 73 Fallow Court Avenue, North Finchley, London N12 0BE (GB). MILNER, Richard [GB/GB]; 5 Richmond Close, Bishops Stortford, Hertfordshire CM23 4PG (GB). SHELVEY, Michael, Francis [GB/GB]; Hillcroft, 1 Church Cottages, Fangfoss, York YO4 5QJ (GB).		(74) Agent: HOBBS, John, David; Corporate Patents & Trade Marks Department, Smith & Nephew plc, Gilston Park, Harlow, Essex CM20 2RQ (GB). (81) Designated States: AT, AU, BB, BG, BR, CA, CH, CS, DE, DK, ES, FI, GB, HU, JP, KP, KR, LK, LU, MG, MN, MW, NL, NO, PL, RO, RU, SD, SE, US, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: POLYURETHANE FOAMS (57) Abstract A hydrophilic polyurethane foam for use in first aid dressings which comprises residues from an aromatic isocyanate prepolymer and an aliphatic isocyanate prepolymer and in which at least one of said prepolymers contains residues from polyether polyol. A process for the formation of the hydrophilic polyurethane and absorptive devices of hydrophilic polyurethane foam such as wound dressings and sanitary devices.		

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POLYURETHANE FOAMS

The present invention relates to hydrophilic polyurethane foams, blends used to prepare these foams, absorptive devices comprising the foams and methods for their preparation.

There have been several proposals to the use of hydrophilic polyurethane foams, for example as an absorbent material, in hygienic and medical absorptive devices such as sanitary towels, tampons, diapers, incontinence pads and wound dressings. Hydrophilic polyurethane foams, however, are usually more expensive than the conventional cellulosic absorbent materials used in such devices and have therefore not been extensively used in commercial absorptive devices. British Patent No. 1429711 (see also United States Patent Nos. 3812618, 3812619, 3874694, 3889417, 3928138, 3929574 and 4137200) discloses a hydrophilic polyurethane foam formed by reacting with water an isocyanate capped polyoxyethylene glycol prepolymer.

It was found, however, that a large molar excess of water was required to obtain satisfactory foams. Removal of this excess water, for example, by drying, rendered these foams relatively expensive to manufacture.

British Patent No. 2188055 discloses hydrophilic polyurethane foams formed by reacting with water the reaction product of polyisocyanate which has a functionality of greater than 2 and polyalkylene glycol mono alkyl or alkaryl ether. These prepolymers require to be mixed with only relatively low amounts of water, thus obviating the need for an elaborate drying stage. The foams are therefore more economical to manufacture than previously known hydrophilic foams.

The reaction rates of aliphatic isocyanate based polyurethane foam systems tend to be undesirably slow for commercial use unless high levels of ethylene oxide containing residues are incorporated into the reactive system.

The presence of high levels of such residues increases the linear swell value of the foam when hydrated. Whilst this effect may not be deleterious for foam products such as tampons or sanitary towels, high linear swell will adversely affect thin foam products such as wound dressings and in particular dressings such as first aid dressings.

Such dressings need to be able to absorb aqueous materials such as wound exudate and to be produced

quickly and economically to compare favourably with conventional fabric and pad dressings.

The present invention seeks to provide a polyurethane foam which is hydrophilic and has good dimensional stability when produced in thin flat sheets and which can be produced rapidly and economically.

Accordingly the present invention provides a polyurethane hydrophilic foam, suitable for use as first aid dressing which and comprise residues of a aromatic isocyanate, of an aliphatic isocyanate, of a polyoxyalkylene monoether and of a polyoxyalkylene polyol such as a polyether polyol.

In another aspect the present invention further provides an absorptive device which comprises a hydrophilic polyurethane foam of the invention. The absorptive device of the invention is preferably a medical or hygienic device such as a wound dressing eg. a first aid dressing, sanitary towel, diaper, incontinence pad tampon, or the like.

In a further aspect the invention provides a process for preparing a hydrophilic polyurethane foam of the invention which comprises blending at least one aromatic based prepolymer with at least one

aliphatic based prepolymer. An aqueous phase is then added to effect the foaming reaction and the foam formed is allowed to set.

According to an embodiment of the present invention there is provided a hydrophilic polyurethane foam comprising residues of a first isocyanate prepolymer derived from a polyoxyalkylene mono ether and an aromatic isocyanate containing at least two isocyanate groups and residues of a second isocyanate prepolymer derived from a polyoxyalkylene mono ether and an aliphatic isocyanate containing at least two isocyanate groups and in which at least one of said isocyanate prepolymers contains residues from a polyether polyol.

The present invention also provides a hydrophilic polyurethane foam formed by blending at least one of said aromatic isocyanate based prepolymers and at least one aliphatic isocyanate based prepolymers and subsequently adding an aqueous phase to catalyse the foaming reaction wherein at least one of the prepolymers is additionally derived from a polyether polyol.

According to the process of the present invention, control over a wider range of processing

variables and foam properties can be achieved than was hitherto possible.

Processing variables which may be controlled more readily than was previously possible include efficiency of mixing and ease of dispensing, cream time, rise time, gel time and cure time.

Foam properties which may be controlled more readily according to the process of the invention include softness and resiliency, density, cell size and structure, water content and capacity, linear swell on hydration and rate of wicking.

These properties may be achieved by blending the prepolymers over a wide range of ratios of the aromatic based prepolymer to the aliphatic based prepolymer properties. Thus foams displaying excellent water uptake, water capacity and strength have been found. The foams also possess a lower level of extractables. Furthermore they can be formed into thin sheets which show excellent conformability.

Suitably the prepolymer blend should contain at least 10w/w% of the solids weight of aliphatic based prepolymer. Similarly it has been found that the blend should contain at least 10w/w% of the solids weight of

aromatic based prepolymer. Aptly the ratio of aromatic isocyanate to aliphatic isocyanate prepolymers will be from 25-75 to 75:25 weight percent. More aptly the ratio of prepolymers will be about 50:50 weight percent.

The hydrophilic polyurethane foams of the invention can be formed by mixing the blend with a stoichiometric amount of water. It is preferred, however, to mix the blend with a low molar excess of water for example 10% by weight of water. It has been found that this low molar excess of water can be easily absorbed by the hydrophilic foam.

Suitable polyoxyalkylene mono ethers for preparing the prepolymer components of the blend of the present invention, may be polyoxyalkylene monoalkyl or monoalkaryl ethers. Preferably the mono ethers are polyalkylene glycol monoethers.

The alkylene moiety of the mono ethers may contain 1 to 4 carbons.

Preferred polyalkylene glycol mono alkaryl ethers are those in which the alkylene group is ethylene.

Suitable polyalkylene glycol mono alkaryl ethers

include those in which the aryl moiety is phenyl. Preferred ethers are those in which the alkyl moiety contains from 1 to 20 carbon atoms eg. octyl or nonyl.

Suitable polyalkylene glycol mono alkyl ethers for forming the reaction product are those in which the alkyl group contains 1 to 20 carbon atoms. Alkylene favoured ethers are those in which the alkyl group is a methyl group. Another class of preferred polyalkylene glycol mono alkyl ethers are those in which the alkyl group contains 10 to 18 carbon atoms, eg. lauryl or cetyl.

Preferred polyalkylene glycol mono alkyl ethers are those in which the alkylene group is ethylene.

The polyalkylene glycol alkyl or alkaryl ether can suitably have an average molecular weight of 180 to 6000.

Apt ethers are polyethylene glycol mono lauryl ethers having an average molecular weight of approximately 1090 and 360 known as Brij 35 and Brij 30 respectively, available from Honeywell Atlas and polyethylene glycol mono methyl ethers having an average molecular weight of approximately 500 and 5000 known as PEG monomethylether molecular weight 550 and

5000 respectively, available from Aldrich Chemicals.

Suitable polyethylene glycol mono nonyl phenyl ethers are commercially available under the Trade names Antarox CO-320, Antarox Co-990, available from GAF (Great Britain) Co. Ltd. Apt polyethylene glycol mono nonyl phenyl ethers, are Dowafax 9N6 and 9N20 having an average molecular wieght in the range of 400-500 and 1100-1200 respectively and available from K+K Greeft Ltd. Typically the ethylene oxide molecular weight can vary in the range 220 to 2200.

The polyethylene glycol mono alkyl or alkaryl ether used in the invention will normally contain water. It is preferred, however, that the ether contains less than 1% by weight water, to limit the number of urea groups formed in the reaction with the polyisocyanate during the prepolymer formulation.

The polyether polyol residues present in at least one of the blend prepolymers, may be derived from polyhydric alcohols, alkylene polyamines, alkylene amines, cyclic amines, amides and polycarboxylic acids. In addition suitable polyols may be derived from hydrophilic reactants. A particularly suitable hydrophilic reactant is ethylene oxide.

Preferred polyether polyols are derived from ethylene oxide and aliphatic polyhydric alcohols. Suitable alcohols may have from 2 to 6 carbon atoms eg. ethylene glycol, pentaerythritol, propylene glycol, 2,3-butylene glycol, glycerol, 1,5-pentanediol and the like.

The polyether polyols may be derived from the polymerisation of ethylene oxide in the presence of the above mentioned di- or polyfunctional reactants.

Particularly suitable polyether polyols for the preparation of foams of the present invention are polyether triols. Preferred polyether triols are polyoxypropylene (PPG) ether triols, end-capped with polyethylene oxide (PEG). Suitably the PEG may comprise 2 to 30w/w% of the polyether triol. Typically the PEG comprises 5 to 15w/w% of the polyether.

Apt polyoxypropylene ether triols, end-capped with PEG have an average molecular weight of 700 to 7000. Typically the PPG ether triols, end capped with PEG will have an average molecular weight in the range of 3000-3500 eg. Arcol 132 available from Arco Chemical Products Europe.

At least one of the blend prepolymer may further

be derived from a copolymer polyether triol derived from the polyether polyols described herein. A particularly preferred copolymer polyether is based on PPG and PEG blocks. Suitably the PEG may be present in the range of 40 to 75w/w of the copolymer. More suitably the PEG may comprise 45-55w/w%. Preferably the PEG comprises 45w/w% of the copolymer. A preferred copolymer polyether is Voranol CP1421 which has an average molecular weight in the range of 3000-3500 and is supplied by Dow Chemicals Europe.

The ratio of triol equivalents to mono ether equivalents may be varied to alter the processing variables and the foam properties. It has been found that suitably the ratio of triol equivalents to mono ether equivalents is in the range of 1.3:1 to 10:1, more suitably from 1:1 to 5:1 and preferably about 3:1.

The isocyanates used for forming the blend prepolymers will have a functionality of at least 2. Suitably both the aliphatic aromatic isocyanates will have a functionality of less than about 2.5.

Suitable aliphatic polyisocyanates for use in the invention include 4,4'-dicyclohexyl methane di-isocyanate (Desmodur W) which has a functionality of 2.0 and is available from Bayer A.G. and hexamethylene

di-isocyanate.

Suitable aromatic isocyanates for forming the blend prepolymers are polymeric methylene di-isocyanates. Polymeric methylene di-isocyanates comprise a mixture of 4,4'-diphenyl methane diisocyanates and one or more of polymeric homologues. Apt polymeric methylene di-isocyanates are known as suprasec VM 10, VM 20 and VM 50 available from ICI and have a functionality of 2.07, 2.13 and 2.49 respectively. Further aromatic isocyanates which may be used include toluene di-isocyanate, methylene-bis-(4-phenyl isocyanate), 3,3'-bitolylene-4,4'-di-isocyanate, 1,4-phenylene di-isocyanate, naphthalene-1,5-di-isocyanate and the like.

A favoured blend for forming foams of the present invention comprises an aliphatic based prepolymer derived from polyoxyethylene glycol mono-nonyl phenyl ether, a polyoxypropylene ether triol, end-capped with PEG, a copolymer polyether triol and 4,4'-dicyclohexyl methane di-isocyanate (Desmodur W). The blend further comprises an aromatic based prepolymer derived from a polyoxyethylene glycol mono-nonyl phenyl ether, a polyoxypropylene ether triol, end-capped with PEG and a polymeric methylene di-isocyanate comprising a

mixture of 4,4'-diphenyl methane di-isocyanate and one or more polymeric homologues.

In forming the prepolymers, the isocyanate and reactive hydrogen containing compound are present in an amount to ensure that the prepolymers contain an excess of isocyanate groups. Aptly the isocyanate to hydroxyl ratio (NCO:OH) is at least 2:1. Suitably the NCO:OH ratio should be less than 6:1. Typically the NCO:OH ratio is in the range of 2.7:1 and 4:1.

The prepolymers employed in the invention contain an excess of isocyanate groups. Suitably they contain an excess of at least 2 w/w% NCO groups, more suitably upto 12%w/w excess NCO groups. Typically the prepolymers contain an excess of 5 to 7w/w% NCO groups.

Normally the mono ethers and polyols will be pre-dried to a water content of less than 1%.

The blend can be reacted with an aqueous phase to form a hydrophilic polyurethane foam of the invention. The hydrophilic polyurethane so formed will normally be a cross-linked hydrophilic polyurethane foam. The hydrophilicity of the foam is believed to be dependent on the oxyethylene groups. Varying the weight % of oxyethylene groups in the constituents of the blend,

can provide the hydrophilic polyurethane foams of the invention with a wide range of water absorption properties. Suitably the hydrophilic foam will absorb at least 5% by weight of its weight of water. The water absorption of the hydrophilic polyurethane foams aptly range from 25% to 95% by weight of polymer. Preferred hydrophilic polyurethane foams of the invention, however, have a water absorption of 50% to 92% by weight of polymer.

The water absorption of the foam is determined by weighing a 1cm cube of the foam, then immersing the foam in water (at 20°C) for 24 hours, removing excess water by lightly blotting the foam with absorbent paper and then re-weighing the foam cube. The water absorption of the foam (% by weight) can then be calculated as

$$\frac{\text{weight of wet foam (g)} - \text{weight of dry foam (g)}}{\text{weight of wet foam}} \times 100$$

The hydrophilic polyurethane foam of the invention will normally be an open cell foam. The open cell foam can suitably have a density of 20 to 350 Kg/m³ and can preferably have a density of 4 to 150Kg/m³.

The hydrophilic polyurethane foam can be in a sheet, moulded or particulate form.

The hydrophilic polyurethane foams of the invention can be used in absorptive devices for example as an absorbent component thereof.

If desired, the prepolymer forming reaction may be catalysed. Suitable catalysts include Dioctyl tin dilaurate (Metatin 812 ES); dioctyl tin mercaptide ester (Metatin 813 and Metatin 713); dibutyl tin dilaurate (T-12); stannous octate (T-9) and Bismuth neodecanoate (Coscat 83). Suitably the catalysts may be added in the range of 0.5 to 0.001w/w %.

Suitably the aqueous phase may contain a catalyst to increase the rate of reaction.

It has been found that a suitable amount of water required to be added to the prepolymer blend can be the stoichiometric amount of water needed to react with the NCO groups in the prepolymers. It is preferred, however, in order to obtain a homogeneous mixture of water and prepolymers to use up to 12% eg. 4 to 10% by weight of water and preferably 5% by weight water in the process.

A suitable catalyst for the reaction is an alkali metal carbonate such as potassium carbonate which can be present in amounts of 0.5 to 1.5% by weight of the blend.

In the process of the invention water or an aqueous solution will normally be provided in liquid form which is mixed and reacted with the prepolymer blend. The water in the process, however, can also be provided by a material such as a metal salt hydrate which releases water in liquid or vapour form when heated. Suitable metal hydrates for use in the invention include $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$, $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$, $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ and $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ which is preferred. In the process the metal salt hydrate which is preferably in particulate form is mixed into the prepolymer blend. The mixture can then be heated to a suitable temperature to release the water for reaction with the prepolymer blend.

The foam can be formed into a sheet or a desired shape by casting the foaming mixture into a release carrier or into a shaped mould and allowing the mixture to rise and set.

The foams produced by the process of the invention can then be incorporated into absorptive

devices using conventional methods. In particular the hydrophilic foams of the present invention may be used in the preparation of first aid dressings since they can be used to form thin flexible and conformable sheets which are ideally suited for making such dressings.

Aptly the polyurethane foam composition of the invention may cast into first aid dressings, for example as disclosed in WO91/01706 and WO91/01707. Suitably such dressings may be cast to thicknesses of 0.5 to 20mm, more aptly from 0.75 to 4mm.

The invention will now be illustrated by reference to the following example:

ALIPHATIC PREPOLYMER FORMULATION

Polyoxypropylene (PPG) ether triol, 2 equiv.
polyethylene oxide (PEG) end-capped (Arcol 1132)

Polyoxyethylene mono-nonyl phenyl 1 equiv.
ether (Dowfax 9N20)

Copolymer polyether triol (Voranol CP 1421) 1 equiv.

4,4'-dicyclohexylmethane di-isocyanate
(Desmodur W) to give an NCO:OH ratio of 3.0:1.

Tin catalyst
(Metatin 812ES, Acima Chemicals Ltd) 0.05 w/w%

AROMATIC PREPOLYMER FORMULATION

Arcol 1132 2 equiv

Polyoxyethylene mono-nonyl phenyl ether 1 equiv.
(Dowfax 9N6)

Methylene diphenyl di-isocyanate
(VM10) to give an NCO:OH ratio of 3.5:1

Tin catalyst (Metatin 812ES) 0.01 w/w%

The aliphatic prepolymer is synthesised by first heating the triols and mono ether in an oven at 60°C to melt them. The melted triols and mono ether are then added to a 700ml flange flask followed by the di-isocyanate. The flask is fitted with an air-driven anchor stirrer, a lid, a dry nitrogen blanket and placed in a water bath at 60°C. The components are stirred vigorously until homogenous. The tin catalyst is added via a disposable syringe, while the contents of the flask are being stirred continuously. The water bath is adjusted to 90°C and the reaction is allowed to continue for 60 minutes until completion, as indicated by the subsiding exotherm. After completion, the prepolymer is poured while still warm into an air-tight jar.

The aromatic prepolymer is formed by following the same steps as indicated above for preparation of the aliphatic prepolymer, except that after adjusting the water bath to 90°C, the reaction is allowed to proceed for 30 rather than 60 minutes.

The aliphatic and aromatic prepolymers are then blended. When a homogenous prepolymer blend is obtained the aqueous phase is added to effect foaming.

The foam formed possesses all the properties discussed above which render it ideally suited for use in absorptive devices, particularly first-aid dressings.

Claims

1. A hydrophilic polyurethane foam, which comprises residues of an aromatic isocyanate, an aliphatic isocyanate, a polyoxyalkylene mono ether and a polyether polyol.
2. A hydrophilic polyurethane foam, which comprises residues of a first isocyanate prepolymer derived from a polyoxyalkylene mono ether and an aromatic isocyanate containing at least two isocyanate groups and residues of a second isocyanate prepolymer derived from a polyoxyalkylene mono ether and an aliphatic isocyanate containing at least two isocyanate groups and in which at least one of said prepolymers contains residues from a polyether polyol.
3. A foam according to claim 1, wherein the polyether polyol is a polyoxyalkylene polyol.
4. A foam according to any of claims 1 to 3, wherein the polyether polyol residues in at least one of the blend prepolymers is derived from hydrophilic reactants.

5. A foam according to any of claims 1 to 4, wherein the polyol residues are polyether triols.
6. A foam according to claim 5, wherein the polyether triol is polyoxypropylene (PPG) ether triols, end-capped with polyethylene oxide (PEG).
7. A foam according to claim 6, wherein the PEG comprises between 2 to 30 w/w% of polyether triol.
8. A foam according to claim 6 or claim 7, wherein the PPG ether triol, end-capped with PEG has a molecular weight between 700 to 7000.
9. A foam according to any of claims 6 to 8, wherein the PEG is present between the ranges of 40 to 75 w/w% of copolymer.
10. A foam according to any of the preceding claims, wherein the ratio of polyol equivalents to mono ether equivalents is between 1.3:1 to 10:1.
11. A foam according to any of the preceding claims, wherein the isocyanates have a functionality of at least 2.

12. A foam according to any one of claims 2 to 11, wherein the aliphatic based prepolymer is derived from polyoxyethylene glycol mono-nonyl phenyl ether, a polyoxypropylene ether triol, end-capped with PEG, a copolymer polyether triol and an aliphatic di-isocyanate.

13. A foam according to claims 2 to 12, wherein the aromatic based prepolymer is derived from a polyoxyethylene glycol mono-nonyl phenyl ether, a polyoxypropylene ether triol, end-capped with PEG and a polymeric methylene di-isocyanate.

14. A foam according to any of claims 2 to 13, wherein the prepolymers contain an excess of isocyanate groups.

15. A foam according to claim 14, wherein the prepolymers contain an excess of at least 2 w/w% NCO groups.

16. A process for the preparation of a hydrophilic polyurethane foam which comprises reacting at least one of each of an aromatic isocyanate, an aliphatic isocyanate, a polyoxyalkylene mono ether and a

polyether polyol to form a foam precursor and thereafter foaming the precursor.

17. A process according to claim 16, in which the foam precursor is a single prepolymer derived from an aromatic isocyanate, an aliphatic isocyanate, a polyoxyalkylene mono ether and a polyether polyol.

18. A process according to claim 16, in which the foam precursor comprises a mixture of isocyanate prepolymers derived from the aromatic isocyanate, the aliphatic isocyanate, the polyoxyalkylene mono ether and the polyether polyol.

19. A process for the preparation of hydrophilic polyurethane foam comprising the steps of:-

- blending at least a first isocyanate based prepolymer and at least a second isocyanate based prepolymer

- adding an aqueous foaming phase wherein at least one of the prepolymers is derived from polyoxyalkylene polyol.

20. A process according to claim 19, wherein the prepolymer blend contains at least 10 w/w% of solids

weight of the first isocyanate based prepolymer and 10 w/w% of solids weight of the second isocyanate based prepolymer.

21. A process according to claim 19 or 20, wherein the ratio of the first isocyanate based prepolymer to the second isocyanate prepolymer is between 25:75 to 75:25 weight per cent.

22. Process according to any of claims 19 to 21, wherein the prepolymer blend is mixed with 10% by weight of water.

23. A foam produced by the process as claimed in any of claims 16 to 22 and having a composition as claimed in any of claims 1 to 15.

24. An absorptive device comprising a foam as claimed in any of claims 1 to 15.

25. An absorptive device according to claim 24 in the form of a first aid dressing.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 92/01527

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC Int.Cl. 5 C08G18/28; C08G18/48; C08G18/72; A61L15/00 C08G18/10		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
Int.Cl. 5	C08G ; A61L	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹		
Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
Y	EP,A,0 299 122 (SMITH&NEPHEW) 18 January 1989 see claims 1-18; examples 17,18 & GB,A,2 188 055 cited in the application ---	1
Y	BE,A,670 906 (ELEKAL) 14 April 1966 see page 2, line 14 - page 4, line 12; claims 1-10; examples 1-6 ---	1
A	US,A,4 292 412 (WOOD) 29 September 1981 see column 3, line 36 - column 12, line 2; claims 1-5 ---	1-25
- / ---		
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>¹⁰ Special categories of cited documents : ¹⁰</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 45%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p> </div> </div>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
29 OCTOBER 1992	11. 11. 92	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	BOURGONJE A.F.	

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	US,A,4 439 553 (GUTHRIE ET AL) 27 March 1984 see column 2, line 24 - column 6, line 59; claims 1-12; examples 1-3 ---	2
A	US,A,3 457 203 (COHEN ET AL) 22 July 1969 see claims 1,2 -----	1,13

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO. GB 9201527
SA 63426**

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information. 29/10/92

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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BE-A-670906	14-04-66	CH-A- 433740	
		DE-B- 1282950	
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		SE-A- 8304496	18-07-84
US-A-3457203	22-07-69	None	